# (19) World Intellectual Property Organization

International Bureau





(43) International Publication Date 2 October 2008 (02.10.2008)

(10) International Publication Number WO 2008/117005 A1

- (51) International Patent Classification: CO2F 9/00 (2006.01) E21B 21/06 (2006.01)
- (21) International Application Number:

PCT/GB2008/000175

English

- (22) International Filing Date: 18 January 2008 (18.01.2008)
- (26) Publication Language: English
- (30) Priority Data: 0706007.2 28 March 2007 (28.03.2007) GB
- (71) Applicant (for all designated States except US): M-I L.L.C. [US/US]; 5950 North Course Drive, Houston, Texas 77072 (US).
- (72) Inventors; and

(25) Filing Language:

(75) Inventors/Applicants (for US only): MCCOSH. KAREN [GB/GB]; M-I Swaco, Aberdeen Research And Technology Center, Ashleigh House, 1 Abbotswell Road, West Tullos, Aberdeen AB12 3AD (GB). PHIPPS, JONATHAN [GB/GB]; 79 School Lane, Lower Cambourne, Cambridge Cambridgeshire CB23 5DX (GB). WAY, PAUL [GB/GB]; 41 The Lane, Hauxton, Cambridge Cambridgeshire CB22 5HP (GB).

- (74) Agents: GILL, Stephen et al.; Mewburn Ellis LLP, York House, 23 Kingsway, London, Greater London WC2B 6HP (GB).
- (81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.
- (84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, NO, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

#### Published:

with international search report

#### (54) Title: PROCESS FOR SEPARATION OF WATER FROM SLOP MUD

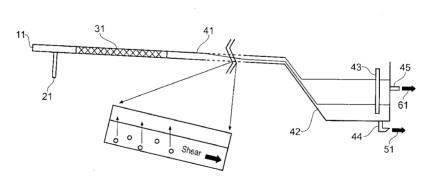


Fig. 1

(57) Abstract: A process for treating slop mud comprises the steps of: mixing a demulsifier into a flow of said slop mud, flowing the mixed slop mud and demulsifier into a flow channel in which the slop mud gravity stratifies into a flow of a water-continuous phase and a flow of an oil-continuous phase, and removing the water-continuous phase from the stratified slop mud.



15

20

25

30

PCT/GB2008/000175

### PROCESS FOR SEPARATION OF WATER FROM SLOP MUD

1

### Field of the Invention

The present invention relates to the separation of slop mud into an oil-continuous phase and a water-continuous phase.

#### Background of the Invention 5

As a result of rigorous environmental regulations shifting towards zero-discharge, drilling wastes are the focus of attention in the oil and gas exploration industry. Drilling with oil-based mud (OBM) or synthetic-based mud (SBM) generates waste streams often referred to as "slop mud" or "slop water". Slop mud is produced when an oil/synthetic/diesel-based drilling fluid becomes contaminated with water. Slop muds may be generated during the drilling process itself if significant quantities of formation water invade the drilling fluid. Alternatively these waste streams may be by-products of cleaning the drill floor, shaker room, pump room and other areas where spillage and interfaces during displacement occur. Contamination can also take place during boat cleaning operations, pit cleaning and other similar operations.

Oil-based drilling fluids are generally used in the form of invert emulsion muds, consisting of an oleaginous phase, a non-oleaginous phase and finely divided particles. typically included are emulsifiers and emulsifier systems, weighting agents, fluid loss additives, viscosity regulators and the like, for stabilizing the system as a whole and for establishing the desired performance properties. Typically, the oil-water ratio (OWR) of a drilling fluid may be in the range 60/40 to 90/10.

After contamination, the slop mud may contain 50% to 90% loosely emulsified water and 10% to 50% non-aqueous drilling

10

15

20

25

30

fluid. The lowering of the oil-water ratio (OWR) increases viscosity, decreases emulsion stability and ultimately renders the fluid unusable as a drilling fluid. However, hydrocarbon contamination renders the slop mud ineligible for, e.g. discharge at sea. Thus the slop mud is typically sent for disposal or reconditioning. For operators, these volumes add up to high disposal expenses and represent a potentially serious environmental issue.

In addition to good fluid design and solids-control equipment to help reduce the amount of waste generated, separation processes exist to treat slop mud waste streams. These function by breaking the weakly emulsified water phase and recovering the OBM/SBM so that mud can be reused without substantial reconditioning. At the same time, the amount of waste generated can be reduced. The primary goal of this form of slop separation is to break only the weakly emulsified water phase and recover the oil/synthetic/diesel-based drilling fluid.

Batch processes have been developed for separating water from slop mud, for example as described in US 2004/0094483 Al and US 2006/0186056 Al.

In a batch process, slop mud is pumped into large stirred treatment tanks to which demulsifying chemicals are added which cause the water droplets to coalesce. The mixing of the demulsifier with the slop mud is inefficient in batch processes due to the volume ratio or "dead spaces" which remain unstirred. Once sufficiently mixed, the stirring is stopped and the slop mud is allowed to separate under gravity. Typically, a water-continuous layer floats to the surface; the oil-continuous layer is denser than water because it contains a significant quantity of dispersed, dense particulate solids that remain oil-wet. Water droplets that have coalesced may have to travel distances of the order of a metre through the

10

15

20

25

30

relatively viscous oil-continuous layer in order to reach the interface between the two layers. Consequently, the separation process can last several hours and batches are often left to separate overnight. In addition to the time required to mix in the demulsifier and to allow the layers to separate, a significant proportion of the process time is taken up by the filling and emptying of the tanks themselves. In order to reduce the time taken for the separation to occur, high doses of demulsifying chemicals may be used. Typically, the demulsifier treatment concentration ranges from 1 to 4% by volume.

Once separation has occurred, the separated constituents (drilling fluid and water) are transferred to separate holding/treatment vessels.

Continuous processes based on centrifuge technology exist for treating an aqueous phase polluted by oil, chemicals and particles in emulsified form, for example slop oil, bilge water and drain water. However, centrifuging results in either two-phase separation of the solids from the liquid phase (oil and water) or three-phase separation of the solids, oils and water. Centrifuging is therefore not adapted to the separation of slop mud, by which it is intended to recover an oil-based or synthetic-based drilling mud which is an oil-continuous phase containing emulsified water and a significant proportion of finely divided particles.

### Summary of the Invention

Faster and more efficient processes for separating excess water from slop mud, to facilitate the recovery and re-use of the drilling fluid and the safe discharge or re-use of the water, would be desirable.

In a first aspect, the present invention provides a process for treating slop mud, comprising the steps of: mixing a

10

15

20

25

30

4

demulsifier into a flow of said slop mud, flowing the mixed slop mud and demulsifier into a flow channel in which the slop mud gravity stratifies into a flow of a water-continuous phase and a flow of an oil-continuous phase, and removing the water-continuous phase from the stratified slop mud.

PCT/GB2008/000175

By maintaining flows of the stratified water-continuous and oil-continuous phases in the flow channel, the process can avoid the long separation times of conventional batch processes. In particular, long travel distances for water droplets through the mud can be avoided.

Typically, the oil-continuous phase is denser than the water-continuous phase. For example, the oil-continuous phase may contain substantially all of the solids content (such as weighting agent) of the slop mud. Thus, the drilling mud can be recovered from the slop mud using this process. This provides an advantage over centrifuge methods of treating waste streams, which separate the solids from the liquid phase(s).

Preferably, the mixed slop mud and demulsifier flow into the flow channel in a continuous stream. This can reduce the space required for storing the slop mud prior to treatment.

Preferably, the water-continuous phase and/or the oil-continuous phase are removed in a continuous stream. An advantage of treating slop mud in a continuous process rather than in a batch process is that the apparatus required can be compact, as large settling tanks are not needed. This is particularly advantageous in off-shore applications.

Preferably, the process may include the further step of heating the slop mud before it flows into the flow channel. An advantage of heating the slop mud is that the rate of separation of the oil-continuous phase and the water-continuous phase may be increased by reducing the viscosity of

the fluids. This increases the overall rate at which slop mud can be treated, and may reduce the concentration of demulsifier required. Reducing the concentration of demulsifier lowers the concentration of contaminants in the recovered water and eases the downstream polishing of the water to meet discharge criteria.

5

10

15

20

25

30

The process may include the further step of mixing a defoamer into the slop mud before the slop mud stratifies. The defoamer may reduce foaming of the separated water-continuous phase caused by, for example, the demulsifier.

Preferably, the flow channel is inclined. In this way, the flows of the water-continuous and oil-continuous phases in the channel can be maintained by gravity. Advantageously, the flow channel is adapted to maintain laminar and stratified flows of the water-continuous and the oil-continuous phases and thereby prevent the separated phases from remixing. Thus, preferably the channel is adapted to maintain a Reynolds number of 2000 or less in the flows of both the water-continuous and oil-continuous phases. Furthermore, the channel may be adapted to maintain a Froude number of 1 or less in the flows of both the water-continuous layers.

The process may include the further step of holding the stratified slop mud in a holding vessel, the water continuous phase and the oil-continuous phase being removed from the holding vessel in two separate flow streams. Preferably, the position of the interface between the oil-continuous phase and the water-continuous phase in the holding vessel is controlled by controlling the rate of flow of one or both of the separate flow streams. The holding vessel may have a sloped side down which the stratified slop mud flows into the vessel. This reduces remixing of the stratified phases.

The mixed slop mud and demulsifier may be flowed through a plurality of flow channels. For example, the plurality of flow channels may be formed by the plates of a lamella separator. Such a separator may also function as a holding vessel for the separated phases. The holding vessel may include a level sensor to determine the position of the interface between the oil-continuous phase and the water-continuous phase.

5

10

15

20

25

30

The demulsifier may contain, for example, an alkyl ethoxylate, alkyl glucoside, branched alkyl sulphosuccinate, octyl or decyl polyglucoside, alkane sulphonate or alkane sulphate. Preferably, the demulsifier contains sodium di(1,3 dimethylbutyl) sulphosuccinate, 2-ethyl hexanol ethoxylate, octyl monoglucoside, or undecyl glucoside. More preferably, undecyl glucoside is used, because this demulsifier has been found to reduce the separation time of the slop mud.

The process may include the further step of reconditioning the oil-continuous phase to produce an oil-based mud or synthetic-based mud suitable for reuse as a drilling fluid. The step of reconditioning the oil-continuous phase may include adding an emulsifying agent and/or further OBM or SBM to the oil-continuous phase.

The water-continuous phase may be removed to a holding tank. The process may comprise the further step of treating the water-continuous phase to remove residual hydrocarbons and/or solids from the water-continuous phase. The further step of treating the water-continuous phase may include adding a defoamer to the water-continuous phase. The further step of treating the water-continuous phase may include centrifuging and/or filtering the water-continuous phase to remove residual hydrocarbons and/or solids. The further step of treating the water-continuous phase may include, for example, the addition of de-oiling polymer, coagulant or flocculant, pH control,

10

15

20

25

30

and/or dissolved air flotation. Suitable flocculant includes inorganic flocculant based on bentonite, aluminium sulphate and/or calcium hydroxide, organic flocculant based on a cationic polymer (e.g. polyacrylamide), a deoiling polymer based on e.g. polyaluminium hydroxychloride, or a combination of any one or more of the above. The water-continuous phase may be heated before the further step of treating the water-continuous phase (e.g. by centrifuging), preferably to a temperature between 40°C and 50°C.

A further aspect of the invention provides an apparatus for treating slop mud, comprising: supplies of a slop mud and a demulsifier, a mixer operatively connected to the supplies for mixing the slop mud and demulsifier, a lamella separator arranged to receive the mixed slop mud and demulsifier, an outlet (e.g. overflow) from the lamella separator for a watercontinuous phase separated from the mixed slop mud and demulsifier, and an outlet (e.g. underflow) from the lamella separator for an oil-continuous phase separated from the mixed slop mud and demulsifier. Preferably, the apparatus further comprises a reconditioning unit for reconditioning the oilcontinuous phase to produce an oil-based mud or syntheticbased mud suitable for reuse as a drilling fluid. Preferably, the apparatus further comprises a treatment unit for treating the water-continuous phase to remove residual hydrocarbons and/or solids from the water-continuous phase.

A further aspect of the invention is the use of a lamella separator to separate a mixture of slop mud and a demulsifier into a denser oil-continuous phase and a less-dense water-continuous phase.

## Brief Description of the Drawings

Embodiments of the invention will now be described by way of example with reference to the accompanying drawings in which:

Figure 1 shows a schematic representation of a process according to a first implementation; and

Figure 2 shows a schematic representation of a process according to a second implementation.

### 5 Detailed Description

10

15

30

A first implementation of the invention will be described with reference to Figure 1. The slop mud enters the system through an inlet 11. The slop mud may be pumped into the system, or it may flow under gravity alone. It then passes through an in-line mixer 31, in which it is mixed with a quantity of demulsifier. The mixer may be a simple static mixer (e.g. a static shear mixer manufactured by Komax or Westfall), or it may be a dynamic one. Alternatively, the action of e.g. a centrifugal pump used to move the slop may be sufficient to mix the demulsifier and slop mud.

In Figure 1, the demulsifier is introduced into the slop mud flow through an inlet 21 prior to entering the mixer 31. Alternatively, the demulsifier may be introduced directly into the mixer.

As a result of the action of the demulsifier, the fluid exiting the mixer will begin to separate. The mixed slop mud and demulsifier flow along an inclined conduit 41, during which the slop mud stratifies due to gravity. The denser oil-continuous phase, which contains substantially all of the solids content of the slop mud, settles towards the bottom of the conduit 41. The less dense, water-continuous phase floats to the surface.

The demulsified slop mud separates rapidly into layers provided that the distance travelled by coalesced water droplets to the interface between the layers is kept relatively small. Therefore the depth of the channel or the

10

15

25

30

diameter of the pipe is preferably not too large, in order to minimise the distance that coalesced drops need to travel in order to reach the water-continuous layer. The length of conduit 41 is preferably sufficient to ensure good separation of the phases.

The conduit 41 may be inclined gently downwards in order to facilitate the flow by gravity. The shear forces between the channel and the flowing slop mud facilitate the separation of the oil-continuous and water-continuous phases by causing gentle mixing. The velocity of the flow must be low enough to allow coalesced droplets to migrate out of the oil-continuous layer and into the water-continuous layer without substantial remixing of the layers. Thus, in each layer, the Reynolds number of the flow is preferably less than 2000 and the Froude number is preferably less than 1 to maintain laminar and stratified flow. These dimensionless numbers are defined as follows:

Reynolds number =  $\rho v l / \eta$ 

Froude number =  $(v^2/q1)^{1/2}$ 

with  $\rho$ =fluid density, v=fluid velocity,  $\eta$ =fluid viscosity, l=layer thickness and g=acceleration due to gravity.

In Figure 1, the stratified flow exits the conduit 41 into a holding vessel 42. To maintain a clean separation of the flowing layers, one side of this vessel is sloped; the layers flow down this slope into the vessel preventing substantial remixing. Exit ports 44, 45 are located near the bottom and top of the vessel respectively. Alternatively, a plurality of exit ports may be provided at various heights. A suitable sensor 43 for detecting the interface between the two fluids is located in the vessel, which may be, for example, a simple float device, conductivity probe or guided wave radar probe. A control loop maintains the position of this interface in the

centre of the vessel by adjusting the flow rates of each layer out of the exit ports.

The outflow 51 of the oil-continuous layer contains substantially all of the solids content of the slop mud and can have an oil-water ratio (OWR) suitable for reuse as a drilling fluid. The oil-continuous phase may therefore be sent directly for re-use as a drilling fluid, or for suitable re-conditioning if necessary. For example, emulsifiers or fresh drilling mud may be added to the recovered oil-continuous phase to reduce the demulsifier concentration and thereby improve the stability of the emulsion.

5

10

15

20

25

30

The water-continuous layer may contain residual hydrocarbons, solids or other contaminants which must be either removed or reduced to acceptably low concentrations for discharge. These remaining contaminants are generally such small droplets or particles that gravity is not sufficiently strong to remove them. Therefore, the outflow 61 from the water-continuous layer may be sent for further treatment in order that the water meets the regulation standards for discharge. outflow 61 may be fed or pumped into a holding tank ready for further treatment. Further treatment of the water-continuous phase may include, for example, centrifuging (in e.g. a disk stack centrifuge), the addition of de-oiling polymers, coagulants or flocculants, pH control, dissolved air flotation, or filtration. Suitable flocculants include inorganic flocculants based on bentonite, aluminium sulphate and calcium hydroxide, organic flocculants based on low and high molecular weight cationic polymers (e.g. polyacrylamide), or deciling polymers based on polyaluminium hydroxychloride, or combinations of the above. The water-continuous layer may be heated, e.g. before centrifuging, to aid separation by reducing viscosity. A temperature of about 45°C is preferred, to prevent scaling.

The process can separate slop mud emulsions using simple demulsifiers such as alkyl ethoxylates, alkyl glucosides and branched alkyl sulfosuccinates. Preferred demulsifying surfactants include sodium di(1,3 dimethylbutyl) sulphosuccinate, 2-ethyl hexanol ethoxylate, octyl monoglucoside, and undecyl glucoside. More preferably, the demulsifier is undecyl glucoside. However, this list of demulsifiers is not exhaustive and other suitable demulsifiers may also be used, for example combinations with alkyl polyglycosides (e.g. octyl and decyl polyglucosides), alkane sulphonates and alkane sulfates. An important factor in determining the choice of demulsifier is the rate of separation of the oil-continuous and water-continuous phases. In order to operate the process continuously, it is preferable that the demulsifier rapidly separates the oil-continuous and water-continuous phases. Preferably, the demulsifier is oilinsoluble to reduce the carry-over of demulsifier into the recovered oil-continuous phase. Of the chemicals listed above, all are oil-insoluble except for 2-ethyl hexanol ethoxylate, which is dispersible in oil.

5

10

15

20

25

30

The inventors have discovered that, provided that vigorous mixing conditions are used, addition of a suitable demulsifying surfactant causes rapid destabilisation of the slop mud emulsion and coalescence of the water droplets within it. Such mixing conditions may lead to the continual break-up and reformation of droplets, but they do not cause reemulsification. Thus the need for gentle mixing conditions is eliminated and a high-energy mixer can be used.

When using undecyl glucoside (Simulsol SL11W<sup>TM</sup>, obtained from Seppic), the inventors found that the rate of separation of the oil-continuous and water-continuous phases increases with temperature. Therefore, it is preferable to heat the slop mud before mixing with the demulsifier. The demulsifier may also

be heated. It may be necessary to increase the proportion of demulsifier added to the slop mud, inversely with temperature, in order to obtain a satisfactory phase separation at relatively low temperatures.

Some demulsifiers, for example undecyl glucoside, cause the separated water-continuous phase to froth. A defoamer may be introduced to the slop mud before it flows into the conduit 41, or before it is mixed with the demulsifier, to reduce this effect. Alternatively, a defoamer may be added to the water-continuous phase after removal from the holding vessel 42, to reduce its frothiness before e.g. centrifuging. Suitable defoamers include silicon-based defoamers (e.g. Defoam X EH<sup>TM</sup> from M-I SWACO, which is an aqueous emulsion of diethylpolysiloxane) and fatty-acid-based defoamers (e.g. Defomex 108N<sup>TM</sup> from Lamberti).

A second implementation of the invention will be described with reference to Figure 2. The slop mud enters the system through an inlet 1. The slop mud may be pumped into the system, or it may flow under gravity alone. It then passes through an inline mixer 3, in which it is mixed with a quantity of demulsifier. Alternatively, the demulsifier may be introduced into the slop mud before it enters the mixer. The demulsifier may be introduced into the slop mud from a demulsifier dosing system 2. As in the first implementation, various types of mixer may be used.

20

25

30

Preferred demulsifiers have been discussed above with respect to the first implementation.

The mixed slop mud and demulsifier then flow into a lamella separator 4. Lamella separators are commercially available (e.g. Pan America Environmental slant plate clarifier systems, or Leiblein GmbH lamella separators) and comprise a stack of plates inclined at an angle. Typically this angle ranges from

10

15

20

25

30

45° to 60° to the horizontal. The spacing between the plates is typically 50 to 75 mm (2 to 3 inches). The spaces between the plates provide a plurality of inclined flow channels, which greatly increase the separation rate compared to a single flow channel. In some lamella separator arrangements, the influent is fed into the lamella separator so that it enters each inclined channel at the bottom of each channel. A disadvantage of such arrangements, however, is that the influent meets a counterflow of emerging oil-continuous phase at the bottom of the channel. In other lamella separator arrangements, however, the influent enters each inclined channel a distance above the bottom the channel. For example, the influent may enter each channel a distance from the bottom of the channel which is equal to about 1/3 of the overall length of the channel.

As the slop mud emulsion separates, the denser oil-continuous phase, including substantially all the solids content of the drilling fluid, settles towards the lower plate of each channel and then flows down to the base of the separator. The less dense, water-continuous layer moves in the opposite direction, beneath the ceiling of each channel towards the top of the separator. Preferably, the flow rate into the lamella separator is such that the flow is laminar and the stratified layers do not remix in the flow channels. Preferably, for both the water-continuous and the oil-continuous phases, the Reynolds number is 2000 or less and the Froude number is 1 or less. The demulsified slop mud separates rapidly into oilcontinuous and water-continuous layers because the distance travelled by coalesced water droplets to the interface between the layers within each flow channel is relatively small.

The underflow 5 of the lamella separator 4 is the oil-continuous phase, which can be removed from an outlet at the bottom of the separator. A pump may be used to remove the

10

15

20

25

30

underflow. The overflow 6 of the lamella separator 4 is the water-continuous phase, which can be removed from the top of the separator. The lamella separator 4 may include a level sensor to determine the position of the interface between the oil-continuous phase and the water-continuous phase. The position of the interface can then be controlled by controlling the rates of removal of the overflow and/or the underflow of the lamella separator.

The recovered oil-continuous phase contains substantially all of the solids content of the slop mud and can have an oil-water ratio (OWR) suitable for reuse as a drilling fluid. It may be reconditioned if necessary, as discussed above with respect to the first implementation.

In Figure 2, the overflow 6 is fed or pumped into a holding tank 7 ready for further treatment. As explained above, the water-continuous phase may contain residual hydrocarbons, solids or other contaminants and may therefore require further treatment to meet the regulation standards for discharge. This further treatment may include centrifuging the water-continuous phase in a disk-stack centrifuge 8, resulting in three-phase separation of the water, oil and solids, which can each be removed from separate outlets. The water-continuous layer may be heated before centrifuging in the disk stack, to aid separation by reducing viscosity. A temperature of about 45°C is preferred, to prevent scaling.

As also discussed above, it may be desirable to introduce a defoamer, either to the slop mud flow or to the separated water-continuous phase, to reduce frothing.

An advantage of the process is that it may be operated continuously and separation times reduced. This means that the apparatus required for the process can be relatively compact. The efficiency of the process is increased by using,

for example, in-line mixers, suitable demulsifiers to obtain a high slop mud separation rate, and a lamella separator with its high separation surface area. In contrast, existing batch methods require the slop mud to settle in tanks for some hours.

### Examples:

5

15

20

25

30

Tests were performed using an apparatus as shown in Figure 2. Table 1 shows the results of Tests 1 to 4 which are described below.

For each test described below, the demulsifier used was undecyl glucoside (Simulsol  $SL11W^{TM}$  from Seppic).

A slop mud was prepared in a slop mud preparation tank by mixing 50% volume of M-I VERSAVERT<sup>TM</sup> oil-based drilling mud (the base mud) and 50% volume of fresh water. A shear mixer was used to prepare the slop so as to achieve a stable and homogeneous emulsion. The slop was continually agitated using submersed agitators and thoroughly mixed each day to ensure phase separation did not take place in the preparation tank. Chemical and physical tests (to API standards) were carried out on the base mud and the slop mud and the slop mud temperature was monitored throughout the trial.

The slop mud was next fed into the system of Figure 2 using a Moyno pump. The demulsifier was injected into the slop feed stream downstream of the Moyno pump through a 2" Westfall static shear mixer. The demulsifier dosing system consisted of a gear pump. Two further 2" Komax static shear mixers were used in-line to achieve the mixing energy required for homogeneous dispersion of the demulsifier into the slop mud, although these extra mixers may not be required.

The mixed slop mud and demulsifier were fed into a lamella separator (a "10m3/hr" model from Tipp Umwelt). A Moyno pump

was used to remove the underflow (oil-continuous layer) from the lamella separator prevent build up of the recovered drilling fluid phase in the lamellas and to prevent short-circuiting of the feed.

A flow meter was used to measure the feed flow rate of the slop mud and demulsifier mix. The temperature of the slop mud, the surfactant dosage and the flow rates of the underflow and overflow from the lamella separator were recorded.

### Test 1

- The demulsifier was added to the slop mud at a concentration of 1% vol. The slop mud and demulsifier mix was fed to the lamella separator at a flow rate of 5 m<sup>3</sup>/hr and at a temperature of 37°C. The recovered mud was pumped out from time to time as the underflow of the lamella separator.
- The base mud was prepared with an OWR of 73/27. After addition of the fresh water to the base mud in a 50/50 ratio, the OWR of the resulting slop mud was reduced to 37/63. The recovered mud from the lamella underflow was found to have an OWR of 75/25 which is comparable to the OWR of the base mud.
- Similarly, the specific gravity (SG), plastic viscosity (PV) 20 and yield point (YP) of the recovered mud were comparable to the base mud properties. The electrical stability (ES) of the recovered mud decreased to 180V from the base mud ES of 800V. This is because some demulsifier is carried over into the oilcontinuous phase and tends to lower the stability of the 25 emulsion. Although the demulsifiers used are preferably oilinsoluble, the oil-continuous phase contains some water, and therefore some demulsifier since the demulsifier will generally distribute throughout all the water present in the slop mud. It is also possible that oil-soluble emulsifiers 30 present in the original drilling mud cause movement of the demulsifier from the water-continuous phase to the oil-

continuous phase. Emulsifiers or fresh mud can be added to the recovered mud to reduce the demulsifier concentration and increase the emulsion stability.

The overflow from the lamella separator was analysed using a hot spin centrifuge and found to be >99% water, with trace amounts of oil and solids.

# Test 2

5

10

15

20

25

30

The properties of the base mud and slop mud used in Test 2 were the same as in Test 1. The demulsifier was added to the slop mud at a concentration of 1% vol. The slop mud and demulsifier mix was fed to the lamella separator at a flow rate of  $10 \text{ m}^3/\text{hr}$  and at a temperature of  $37\,^{\circ}\text{C}$ .

The underflow of the lamella separator was pumped out continuously at a rate of approximately 4.5m<sup>3</sup>/hr. This rate is close to that predicted for continuous operation if the process yields 90% water recovery from a slop mud comprising a 50/50 mix of mud and fresh water.

The properties of the recovered mud from the lamella separator underflow were acceptably close to those of the base mud. The OWR of the recovered mud was identical to that of the base mud. Again, the ES of the recovered mud decreased from the base mud ES of 800V, to 240V.

The overflow from the lamella separator was analysed using a hot spin centrifuge and found to be >99% water, with trace amounts of oil and solids.

This test demonstrates that the process according to the second implementation of the invention can continuously separate slop mud into an oil-continuous phase and a water-continuous phase at a rate of at least 10 m<sup>3</sup>/hr. The oil-continuous phase has properties very close to those required

WO 2008/117005 PCT/GB2008/000175

for reuse as a drilling mud, and therefore requires very little further conditioning.

### Test 3

5

10

15

20

25

30

The properties of the base mud and slop mud used in Test 3 were the same as in Test 1. The slop mud and demulsifier mix was fed to the lamella separator at a flow rate of  $10\text{m}^3/\text{hr}$  and at a lower temperature of  $21^{\circ}\text{C}$  to investigate the effect of slop mud temperature on the process.

In Test 2, with 1% demulsifier (by volume) at 37°C and a feed flow rate of 10 m³/hr, phase separation of the slop mud was observed to occur as the slop entered the feed chamber of the lamella separator. However, on reducing the temperature to 21°C, a decrease in the phase separation was observed. The demulsifier dose was therefore increased to 1.3% vol. to obtain phase separation as the slop entered the feed chamber.

The underflow of the lamella separator was pumped out continuously at a rate of approximately 7.2m<sup>3</sup>/hr.

The properties of the recovered mud (the lamella separator underflow) were found to be close to those of the base mud and acceptable. The OWR of the recovered mud was 71/29.

#### Test 4

The properties of the base mud and slop mud used in Test 4 were the same as in Test 1. The demulsifier was added to the slop mud at a lower concentration of 0.5% vol. The slop mud and demulsifier mix was fed to the lamella separator at a flow rate of  $10~\text{m}^3/\text{hr}$  and at a temperature of  $37\,^\circ\text{C}$ .

The underflow of the lamella separator was pumped out continuously at a rate of approximately  $4.5 \,\mathrm{m}^3/\mathrm{hr}$ .

The OWR of the recovered mud was 56/44, which was substantially higher than that of the slop mud, 37/63.

However, this value was lower than that of the original base 13/27.

The overflow from the lamella separator was analysed using a hot spin centrifuge and found to contain 92% water, with 5% oil and 3% solids.

These results show that the conditions of Test 4 are not optimum, although a useful quantity of water was removed from the slop mud.

### Further Treatment of the Water-Continuous Phase

A further test was carried out using a slop mud feed rate of 10 10m³/hr, demulsifier Simulsol SL11W1™ at 1.3% concentration by volume, and pumping out the underflow from the lamella separator at 5-6m<sup>3</sup>/hr. Once the process was optimised and steady state conditions were achieved for 30 minutes, the overflow stream from the lamella separator was collected in 15 the holding tank ready for processing it through a disk stack centrifuge. The presence of the undecyl glucoside demulsifier caused significant foaming of water-continuous phase. defoamer Defoam X  $EH^{TM}$  (an aqueous solution of diethyl polysiloxane) was therefore added before disk stack operation. 20 Tests of the disk stack centrifuge were carried out using a feed flow rate from the holding tank of 0.5 m<sup>3</sup>/hr and 3 m<sup>3</sup>/hr. Water samples taken upstream and downstream of the disk stack during the operation were analysed.

The overflow stream of the lamella separator (i.e. upstream of the disk stack) had a total petroleum hydrocarbons (TPH) value of 53 mg/l. The addition of the defoamer at a concentration of 167 ppm increased this value further, to within the range 190-255 mg/l. The disk stack treatment resulted in a steady 68%-77% reduction of the TPH value, and the water recovered downstream of the disk stack had a TPH value of 17 mg/l and 51-79 mg/l, with and without defoamer addition respectively.

The water recovered from the disk stack can be further treated, for example, by filtration, to meet the discharge criteria of <15~mg/l TPH.

			Test 1		Test 2		Test 3		Test 4	
	Base Mud	Slop Mud	Overflow	Underflow	Overflow	Underflow	Overflow	Underflow	Overflow	Underflow
Feed flow rate (m³/hr)			5	5	10	10	10	10	10	10
% vol. demulsifier			1	1	1	1	1.3	1.3	0.5	0.5
Slop mud temperature			37°C	37°C	37°C	37°C	21°C	21°C	37°C	37°C
Underflow pump-out rate			On-off pump	On-off pump	4.5	4.5	7.2	7.2	4.5	4.5
SG	1.25	1.12		1.23		1.2		1.22		1.17
PV	49	98	_	31		30		32		37
YP	18	76		16		12		15		17
Solids % v/v	16	10	Trace	15	Trace	15		15	3	14
Oil % v/v	61	33	Trace	64	Trace	62		60	5	48
Water % v/v	23	57	>99	21	>99	23		25	92	38
OWR	73/27	37/63		75/25		73/27		71/29		56/44
ES	800	160	n/a	180	n/a	240		260	n/a	140

Table 1: Test results

While the invention has been described in conjunction with the exemplary embodiments described above, many equivalent modifications and variations will be apparent to those skilled in the art when given this disclosure. Accordingly, the exemplary embodiments of the invention set forth above are considered to be illustrative and not limiting. Various

10

WO 2008/117005 PCT/GB2008/000175

changes to the described embodiments may be made without departing from the spirit and scope of the invention.

All the references cited herein are incorporated by reference in their entirety.

### CLAIMS

5

20

25

30

 A process for treating slop mud, comprising the steps of: mixing a demulsifier into a flow of said slop mud,

flowing the mixed slop mud and demulsifier into a flow channel in which the slop mud gravity stratifies into a flow of a water-continuous phase and a flow of an oil-continuous phase, and

removing the water-continuous phase from the stratified slop mud.

- 2. The process according to claim 1, wherein the oilcontinuous phase is denser than the water-continuous phase.
  - 3. The process according to any one of claims 1 to 2, wherein the mixed slop mud and demulsifier flow into the flow channel in a continuous stream.
- 15 4. The process according to any one of claims 1 to 3, wherein the water-continuous phase and/or the oil-continuous phase is removed from the stratified slop mud in a continuous stream.
  - 5. The process according to any one of claims 1 to 4, including a further step of heating the slop mud before it flows into the flow channel.
  - 6. The process according to any one of claims 1 to 5, wherein the flow channel is inclined.
  - 7. The process according to any one of claims 1 to 6, wherein the flow channel is adapted to maintain laminar and stratified flows of the water-continuous phase and the oil-continuous phase.
    - 8. The process according to claim 7, wherein the channel is adapted to maintain a Reynolds number of 2000 or less in the flow of the water-continuous phase and the flow of the oil-continuous phase.

- 9. The process according to claim 7, wherein the channel is adapted to maintain a Froude number of 1 or less in the flow of the water-continuous phase and the flow of the oil-continuous phase.
- 10. The process according to any one of claims 1 to 9, comprising the further step of: holding the stratified slop mud in a holding vessel, the water continuous phase and the oil-continuous phase being removed from the holding vessel in separate flow streams.
- 11. The process according to claim 10, in which the position of the interface between the oil-continuous phase and the water-continuous phase in the holding vessel is controlled by controlling the rate of flow of one or both of the separate flow streams.
- 12. The process according to claim 10 or claim 11, wherein the holding vessel has a sloped side down which the stratified slop mud flows into the vessel.
  - 13. The process according to any one of claims 1 to 12, wherein the mixed slop mud and demulsifier are flowed though a plurality of flow channels.

30

- 14. The process according to claim 13, wherein the plurality of flow channels is formed by the plates of a lamella separator.
- 15. The process according to claim 14, wherein the plates of the lamella separator are inclined at an angle of between 45° and 60° to the horizontal.
  - 16. The process according to any one of claims 1 to 15, in which the demulsifier contains sodium di(1,3 dimethylbutyl) sulphosuccinate, 2-ethyl hexanol ethoxylate, octyl monoglucoside, undecyl glucoside.

17. The process according to any one of claims 1 to 16, comprising the further step of reconditioning the oil-continuous phase to produce an oil-based mud (OBM) or synthetic-based mud (SBM) suitable for reuse as a drilling fluid.

5

15

20

30

- 18. The process according to claim 17, wherein the step of reconditioning of the oil-continuous phase includes adding an emulsifying agent and/or further OBM or SBM to the oil-continuous phase.
- 19. The process according to any one of claims 1 to 18, wherein the water-continuous phase is removed to a holding tank.
  - 20. The process according to any one of claims 1 to 19, comprising the further step of treating the water-continuous phase after removal from the stratified slop mud to remove residual hydrocarbons and/or solids from the water-continuous phase.
  - 21. The process according to claim 20, in which the further step of treating the water-continuous phase includes centrifuging and/or filtering the water-continuous phase to remove residual hydrocarbons and/or solids.
  - 22. The process according to claim 20, in which the water-continuous phase is heated before the further step of treating the water-continuous phase.
- 23. The process according to claim 22, in which the water-continuous phase is heated to a temperature between 40°C and 50°C.
  - 24. The process according to any one of claims 1 to 23, including the further step of mixing a defoamer into the slop mud before the mud stratifies.

- 25. The process according to any one of claims 20-23, in which the further step of treating the water-continuous phase includes adding a defoamer to the water-continuous phase.
- 26. An apparatus for treating slop mud, comprising: supplies of a slop mud and a demulsifier,

10

20

25

a mixer operatively connected to the supplies for mixing the slop mud and demulsifier,

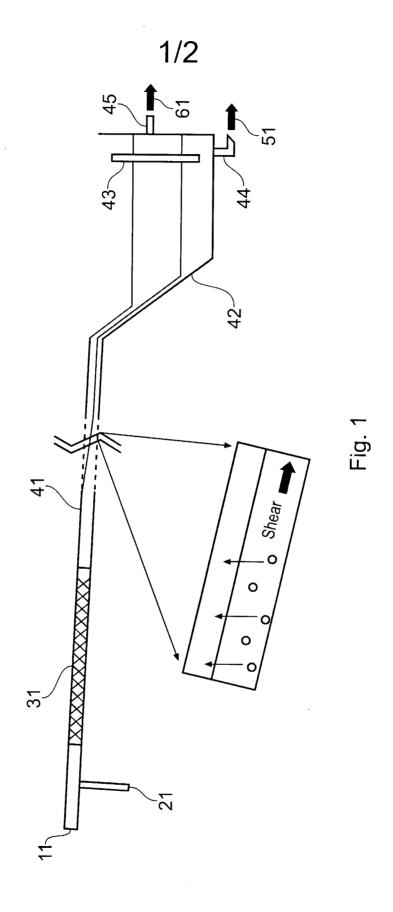
a lamella separator arranged to receive the mixed slop mud and demulsifier,

an outlet from the lamella separator for a watercontinuous phase separated from the mixed slop mud and demulsifier, and

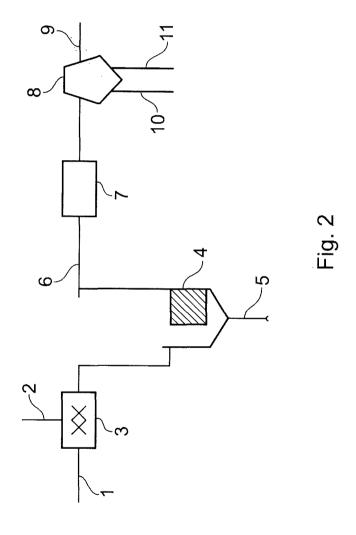
an outlet from the lamella separator for an oil-continuous phase separated from the mixed slop mud and demulsifier.

- 27. The apparatus of claim 26, further comprising a reconditioning unit for reconditioning the oil-continuous phase to produce an oil-based mud or synthetic-based mud suitable for reuse as a drilling fluid.
  - 28. The apparatus of claim 26 or claim 27, further comprising a treatment unit for treating the water-continuous phase to remove residual hydrocarbons and/or solids from the water-continuous phase.
  - 29. The use of a lamella separator to separate a mixture of a slop mud and a demulsifier into a denser oil-continuous phase and a less-dense water-continuous phase.

WO 2008/117005 PCT/GB2008/000175



2/2



### INTERNATIONAL SEARCH REPORT

International application No PCT/GB2008/000175

A. CLASSIFICATION OF SUBJECT MATTER
INV. C02F9/00 E21B21/06

According to International Patent Classification (IPC) or to both national classification and IPC

#### B. FIELDS SEARCHED

 $\begin{array}{ll} \mbox{Minimum documentation searched (classification system followed by classification symbols)} \\ E21B & C02F \end{array}$ 

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

### **EPO-Internal**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 2004/094483 A1 (MUELLER FRANK MANFRED FRANZ [NO]) 20 May 2004 (2004-05-20) cited in the application page 2, paragraph 14 - paragraph 15	1-6,16, 19, 21-23, 26,28,29
<b>Y</b>	US 4 551 239 A (MERCHANT PHILIP [US] ET AL) 5 November 1985 (1985-11-05)  column 1, line 22 - column 9, line 36	1-6,16, 19, 21-23, 26,28,29
<b>A</b>	US 2006/186056 A1 (IVAN CATALIN [US]) 24 August 2006 (2006-08-24) cited in the application the whole document	1-28
	-/ <del></del>	

X Further documents are listed in the continuation of Box C.	X See patent family annex.				
* Special categories of cited documents  *A* document defining the general state of the art which is not considered to be of particular relevance  *E* earlier document but published on or after the international filing date  *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)  *O* document referring to an oral disclosure, use, exhibition or other means  *P* document published prior to the international filing date but later than the priority date claimed	<ul> <li>*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</li> <li>*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</li> <li>*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</li> <li>*&amp;* document member of the same patent family</li> </ul>				
Date of the actual completion of the international search	Date of mailing of the international search report				
27 March 2008	07/04/2008				
Name and mailing address of the ISA/  European Patent Office, P.B. 5818 Patentlaan 2  NL – 2280 HV Rijswijk  Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  Fax: (+31-70) 340-3016	Authorized officer  Morrish, Susan				

## **INTERNATIONAL SEARCH REPORT**

International application No
PCT/GB2008/000175

C(Continua		T.
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Υ	WO 98/08587 A (HYDROTECH DYNAMICS LTD [CA]) 5 March 1998 (1998-03-05) page 10 - page 14	26,29
Y	WO 03/099724 A (CB CONSULT N V [BE]; BURSSENS CARL [BE]) 4 December 2003 (2003-12-04) page 1, paragraph 4	26,29
<b>A</b>	US 4 507 208 A (SIMON JULES A [US] ET AL) 26 March 1985 (1985-03-26) column 1, line 6 - column 3, line 51	1–28
A	US 6 391 195 B1 (LAYTON KENNETH R [CA]) 21 May 2002 (2002-05-21) column 1, line 5 - column 3, line 28	1–28
A	WO 89/02774 A (NOVATEC INC [US]) 6 April 1989 (1989-04-06) abstract	1,26
	<del></del>	
,		
•		
-		

### INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No PCT/GB2008/000175

Patent document cited in search report				Publication date		Patent family member(s)	Publication date	
	US	2004094483	A1	20-05-2004	AU	2003286615		15-06-2004
					CA	2506098		03-06-2004
					EP	1561004		10-08-2005
			•		MX	PA05005189		19-08-2005
					WO.	2004046498	A1 	03-06-2004
	US	4551239	Α	05-11-1985	AU	570723		24-03-1988
					CA	1233390		01-03-1988
					EP	0174399		19-03-1986
		<u> </u>			JP	61066791	Α	05-04-1986
	US	2006186056	A1	24-08-2006	NONE	· ·		
	WO	9808587	Α	05-03-1998	AU	4007597	A	19-03-1998
					CA	2184535	A1	01-03-1998
					US	5814230	A	29-09-1998
2	WO	03099724	Α .	04-12-2003	AU	2003240319	A1	12-12-2003
					ΒĒ	1014852	A7.	04-05-2004
				•	EP	1562866	A1	17-08-2005
	US	4507208	Α	26-03-1985	AU	3154484	Α	07-02-1985
					CA	1241463	A1	30-08-1988
					EP	0148264	A1	17-07-1985
		• •			WO	8500354	A1	31-01-1985
. •	US	6391195	B1	21-05-2002	NONE			
	WO	8902774	Α	06-04-1989	AU	2608088	Α	18-04-1989
					BR	8807229		27-03-1990
					DK	269189	Α	02-06-1989
		•			ΕP	0334944	A1	04-10-1989
	٠			•	JP	2501989	T	05-07-1990